Method for the Production of Conjugated Linoleic Acid

Field of the Invention

This invention relates generally to fatty acids and, more particularly, to a new process for the production of conjugated linoleic acid by saponification of its esters and neutralization with phosphoric acid.

Prior Art

Polyunsaturated linoleic acids with conjugated double bonds, which are commercially available as "CLA" (conjugated linoleic acids), belong to the essential fatty acids for human beings and animals and are therefore used as food additives. Conjugated linoleic acid is normally produced from triglycerides which have a high percentage content of – normally unconjugated – linoleic acid, such as thistle or sunflower oil for example. The triglycerides are isomerized in the presence of basic catalysts and then saponified. A disadvantage in this regard is that, on the one hand, the saponification step yields many unwanted waste materials and, on the other hand, large quantities of alkalis are required, which can quickly result in corrosion in the reactors used. To avoid this, linoleic acid alkyl esters have more recently been used as preferred starting materials and, in a first step, are isomerized to the CLA esters and then saponified. In this process, however, utilization of reactor capacity is often very poor. The profitability of the process is seriously restricted by large volumes of water, poor yields and unwanted secondary products.

Accordingly, the problem addressed by the present invention was to provide a process for the production of conjugated linoleic acid which would be distinguished by very high profitability and which would lead to an end product in high yields and purity.

Description of the Invention

The present invention relates to a process for the production of conjugated linoleic acid, in which

(a) linoleic acid lower alkyl esters are isomerized in the presence of alkali metal alcoholates,

(b) the now conjugated linoleic acid lower alkyl esters are saponified with water in the presence of lye and

(c) the saponification product is neutralized with phosphoric acid.

It has surprisingly been found that the neutralization of a saponification product of conjugated linoleic acid lower alkyl esters with phosphoric acid leads to very good utilization of reactor capacity in the production of conjugated fatty acids. Reverse esterification after the saponification step is minimized, so that few unwanted secondary products are formed during the production process. After neutralization with phosphoric acid and subsequent phase separation, an end product is obtained in high yields and high purity by virtue of the low ester content.

Conjugated linoleic acid lower alkyl esters

Starting materials for the process according to the invention are linoleic acid lower alkyl esters which preferably correspond to formula **(I)**:

$$R^1CO\text{-}OR^2 \qquad (I)$$

where R^1CO is the acyl group of a linoleic acid and R^2 is a linear or branched alkyl group containing 1 to 5 carbon atoms. In one particular embodiment, conjugated linoleic acid methyl and/or ethyl esters are used.

Isomerization

The isomerization of the linoleic acid lower alkyl esters is carried out with alkali metal alcoholates in an inert gas atmosphere at temperatures in the range from 90 to 150°C, preferably at temperatures in the range from 100 to 130°C and more particularly at temperatures in the range from 105 to 125°C.

In a preferred embodiment, alkali metal alcoholates containing 1 to 10 carbon atoms are used as bases during the isomerization, potassium methanolate, potassium ethanolate or potassium-t-butylate being particularly preferred.

Saponification

The saponification of the isomerized linoleic acid lower alkyl esters with aqueous lyes is carried out at temperatures in the range from 40 to 90°C, preferably at temperatures in the range from 60 to 80°C and more particularly at temperatures in the range from 65 to 75°C. It is continued to a cleavage level of 80 to 100% by weight and preferably above 98%.

Neutralization

The most important step of the process in terms of process economy (high reactor capacity utilization) is the neutralization with phosphoric acid and working up by phase separation, the salts formed remaining dissolved in the aqueous phase. The phosphoric acid is preferably used in a concentration of 75 to 85% by weight for the neutralization step. The neutralization step is also carried out at temperatures of 40 to 90°C, preferably at temperatures of 60 to 80°C and more particularly at temperatures of 65 to 75°C. Before it is neutralized, the product may be adjusted to the required viscosity by addition of water.

Working up

The neutralization step is followed by phase separation at a temperature of 50 to 100°C and preferably at a temperature of 70 to 90°C. The phase separation is optimized by elevated temperatures. It is followed by drying in vacuo at a temperature above 100°C and preferably at a temperature above110°C.

Example

Production of conjugated linoleic acid from linoleic acid ethyl ester

1190 g linoleic acid ethyl ester were introduced into a heatable flask and 60 g potassium ethanolate (32% by weight) were then added with stirring under nitrogen at a temperature of 110°C, ethanol being continuously distilled off. After addition of 190 g water, 1070 g of a 25% by weight potassium hydroxide solution were pumped into the flask at a temperature of 70°C for saponification. Another 770 g water were then introduced with stirring and 510 g phosphoric acid (85% by weight) were added at a temperature of 70°C for neutralization. Washing water was then removed, followed by phase separation at a temperature of 70 to 90°C.

The conjugated linoleic acid thus obtained had the following characteristics:

acid value:	199
saponification value:	200
OH value:	4.9
iodine value:	162
unsaponifiables:	0.1%